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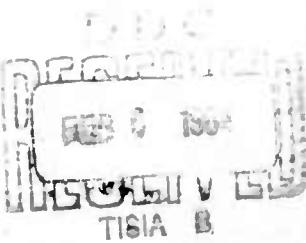
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INVESTIGATION OF LASER HOST MATERIALS

SEMIANNUAL TECHNICAL SUMMARY REPORT

428956



Autonetics A DIVISION OF NORTH AMERICAN AVIATION, INC., ANAHEIM, CALIFORNIA



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INVESTIGATION OF LASER HOST MATERIALS

SEMIANNUAL TECHNICAL SUMMARY REPORT

20 May 1963 Through 31 December 1963

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I. INTRODUCTION

The purpose of this research is to grow laser host crystals of CeO_2 and MgO doped with elements selected from the group Eu, Nd, Pr, Cr, Mn, Co, and V, and to determine the properties of fluorescent spectra, lifetimes, line widths, absorption spectra, band widths of absorption bands, and absorption coefficients of the main peaks.

The cubic class of crystals offers the possibility of improved lifetimes for excited ions in laser crystals. The cerium dioxide crystals of this work have the fluorite structure, and the magnesium oxide is of the sodium chloride structure. Cerium dioxide is of interest because it is a near-rare earth oxide that should permit inclusion of rare earth dopants without severe distortion of the structure. Magnesium oxide crystals are to be grown by a low-temperature chemical vapor technique offering the promise of more nearly perfect crystals, which also should enhance the lifetime of the dopant ion.

Because of the considerable problems associated with growing single crystals of these two materials, this research has been restricted almost exclusively to determining growth conditions.

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II. MgO GROWTH

Magnesium oxide growth utilizes a chemical vapor deposition technique first reported by Cech and Alessandrini(1) for the growth of single crystal films of NiO, CoO, and FeO on MgO substrates. In this process, magnesium halide vapors are hydrolyzed to deposit magnesium oxide on the surface of a seed crystal. The reaction may be written $MgX_2(g) + H_2O(g) \xrightarrow{\Delta} MgO(s) + 2HX(g)$ where X can be Cl, Br, or I. The seeds are MgO crystals supplied by the Norton Company or the Muscleshals Electrochemical Company. The MgO can be doped with certain elements by adding the pertinent halide to the reacting gases by one scheme or another.

A typical arrangement is a straight Vycor tube reactor in a vertical position with $MgCl_2$ supported in a Vycor crucible and the MgO seed suspended 1 in. above the $MgCl_2$. An inert gas flows upward at ~70 ft/hr through the reactor tube. A cylindrical, nichrome wound furnace is used to heat the reactor to 1100 C, at which time H_2O is introduced by bubbling all or part of the incoming carrier gas through H_2O and then into the reactor. Deposition occurs until the $MgCl_2$ supply is exhausted, normally 90 to 120 min. The MgO deposit weighs anywhere from 20 to 150 mgs; with $MgCl_2$ at 1100 C, the rate usually is 70 mgs/hr, giving a deposit of 140 mgs in a 2-hr run.

A. WORK IN SINGLE CRYSTAL GROWTH

Early work centered upon determining conditions for single crystal growth and included the following inter-related subjects:

1. Source Materials and Deposition Temperatures

Anhydrous $MgCl_2$, $MgBr_2$, and MgI_2 were tried as source materials. It soon was discovered that only completely anhydrous, freshly sublimed material consistently produced single crystal deposits. The lowest temperatures which gave single crystal growth were 1075 C for $MgCl_2$, 950 C for $MgBr_2$, and 680 C for MgI_2 ; below these temperatures only polycrystalline deposits or no deposits at all were obtained. Only a few runs were made with MgI_2 since preparation of the anhydrous form was quite tedious and there was no apparent advantage in depositing at the lower temperature. Considerable work was done with both $MgBr_2$ and $MgCl_2$. The latter was preferred because it could be purchased in the anhydrous form and only needed to be vacuum sublimed before it was used.

Presumably, the upper limit for the deposition temperature would be the melting point of the MgO, although this has not been studied exhaustively. Tests with MgBr₂ source material showed that good deposits were obtained when the seed temperature was as high as 1150 C if the seed was placed ~4 in. above the MgBr₂ and the MgBr₂ temperature was raised slightly to 1000 C; this compared to ~970 C for the temperature of both the MgBr₂ and the seed under standard conditions.

2. Seeds

The seeds have been either freshly-cleaved or chemically-polished MgO crystals; in either case growth was on the (100) faces. Surface conditions affected the type of growth observed. Figure 1 shows the spiral growth observed on a freshly-cleaved seed. Figure 2 shows the hillock growth observed on a chemically-polished seed. A few ordered but discontinuous deposits were made using seeds having mechanically-polished (111) faces.



Figure 1. Spiral Growth on Freshly Cleaved Seed
(100 Magnification)

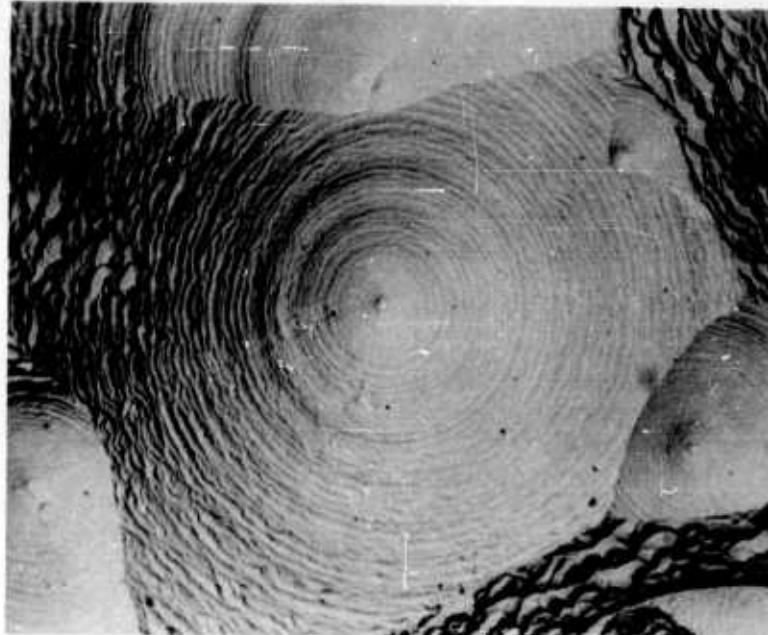


Figure 2. Hillock Growth on Chemically Polished Seed
(100 Magnification)

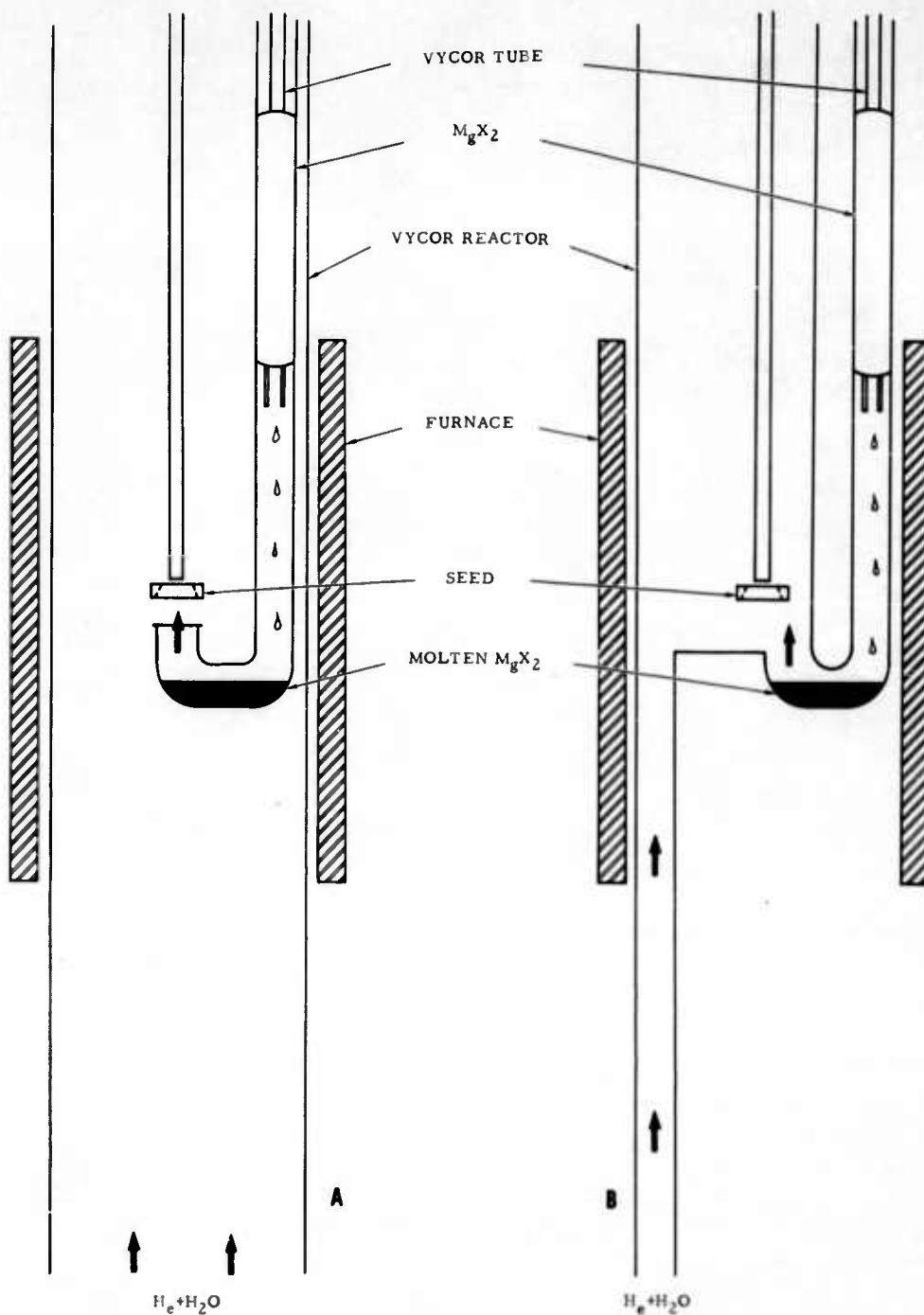
3. Reactor Geometry

Besides the vertical tube arrangement, horizontal and U-tube configurations were used; however, they offered no advantages and, in fact, gave poor reproducibility and uneven deposits. The U-tube configuration was particularly sensitive to gas flow conditions, whereas the standard vertical tube worked successfully over a wide range of inert-gas flow rates and vapor pressure of water. In the vertical tube the critical parameters were MgX_2 temperature, seed temperature, and distance between MgX_2 and seed.

B. LONG RUNS

In order to grow sizable crystals, conditions had to be found for continuing the deposition runs for several hours or even days (at 70 mgs/hr it would take ~51 hr to give a 1 cc crystal).

The best method to replenish the MgX_2 was to feed a solid cylinder of it into one arm of a Vycor U-tube, where it melted and formed a molten pool at the bottom of the U-tube (see Figure 3). This pool was maintained at a temperature to give the desired vapor

Figure 3. Reactors for Continuous Deposition of MgO

pressure of MgX_2 ; the MgX_2 vapors then entered the reactor and reacted with the H_2O at the seed.

The reactors shown in Figure 3 proved to be the most useful, though each reactor had its problems. In the apparatus shown in Figure 3A, the reaction proceeded satisfactorily until MgO crystallites started to build up at the mouth of the MgX_2 source tube; once these crystallites started to form they grew rapidly and prevented the desired growth on the seed. Rounding the edges delayed the crystallite formation only briefly. Bringing the water in by a special tube nearer the seed simply transferred the crystallite growth to that tube. Dry helium gas forced through the U-tube to sweep the MgX_2 vapors toward the seed helped somewhat but did not solve the problem completely.

In the apparatus shown in Figure 3B, the unwanted nucleation was less serious. Here the main problem was the extreme sensitivity to gas flow conditions. Each of these reactors had to be tested with short runs, and flow adjustments had to be made before long runs were conducted.

One other problem was the formation of unwanted polycrystallites on the seed edges. This spurious growth was nearly eliminated by rounding the edges mechanically or chemically, and by rotating the seed during growth.

Recent 8-hr runs in the apparatus shown in Figure 3B have resulted in single crystal deposits weighing 0.5 gm. These were very even growths and indications are that longer runs will give even bigger deposits. Usually one could not differentiate between deposit and substrate when viewing across a cleaved section; however, with some samples certain lighting effects would show the deposit area and, of course, etching would show the deposit area very clearly. Figure 4 shows a view through a cleaved section of a sample that had five successive deposits of ~ 100 mg each to give a final thickness of 0.3 mm over a 4 cm^2 area. While this deposit was visually inferior to those of the same size grown in single runs, it at least could be photographed to give evidence of the crystal size currently obtainable.

Etch pits showed that dislocation counts improved from $>10^7/\text{cm}^2$ on the earliest thin films to $\sim 10^6/\text{cm}^2$ for the thicker films, with some areas approaching $10^5/\text{cm}^2$. The seed crystals, grown from the melt by the carbon arc method, have $\sim 10^5/\text{cm}^2$.

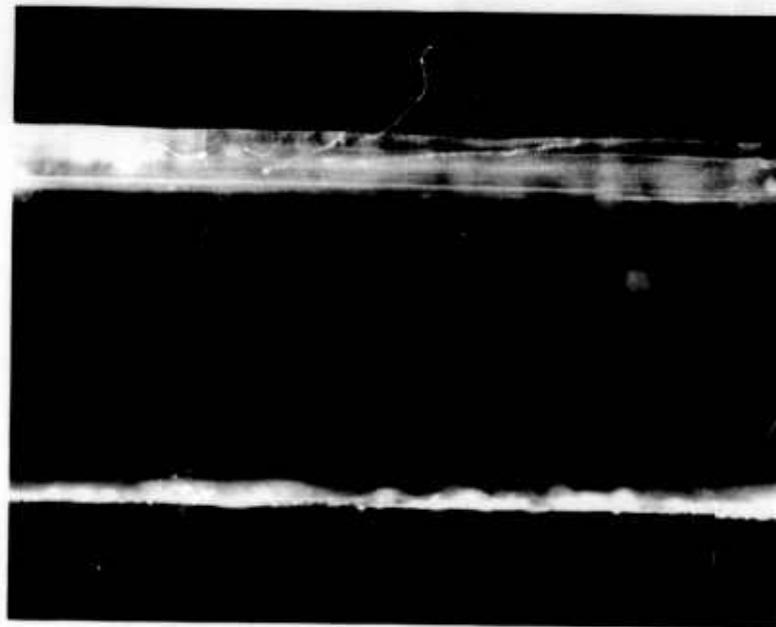


Figure 4. View of Deposit and Substrate Through Cleaved Section (30 Magnification)

Most of the films displayed a very high etch pit count at the substrate-deposit interface; however, in a few instances, the etch pits disappeared completely at the interface, as shown in Figure 5. Figure 5 also demonstrates that substrate imperfections were not necessarily continued into the deposit. (Note that the subgrain boundary in the substrate terminated at the interface.) More stress can be placed on improving crystal quality now that larger crystals are being grown.

C. DOPING

As mentioned previously, the MgO can be doped with certain elements by adding the pertinent halide to the reacting vapors. Factors which must be considered include vapor pressures and stabilities of the available halides, desired oxidation state of the dopant ion, whether the dopant ion will be incorporated readily into the MgO lattice, whether special reducing or oxidizing atmospheres will be required during deposition, and whether the conditions for single crystal growth will be significantly altered.

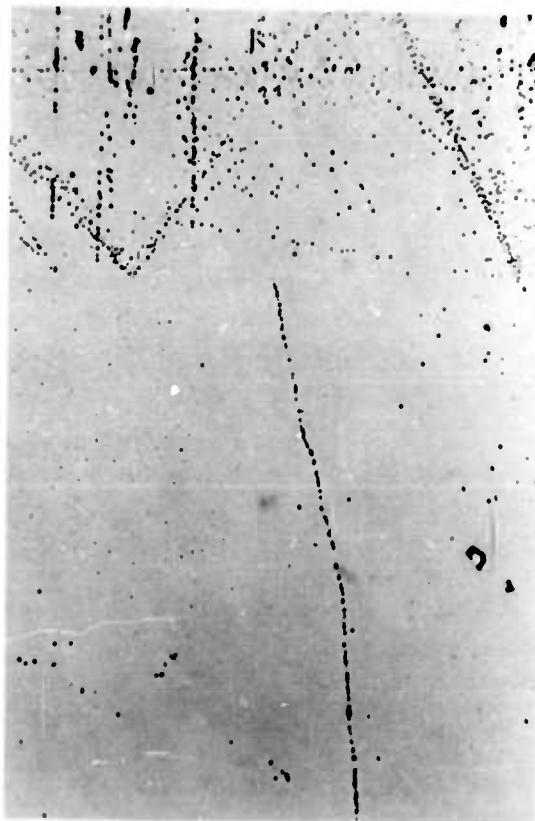


Figure 5. Etch Pattern of MgO Deposit on MgO Substrate
(250 Magnification)

Most of the work in this period has been done with Co^{+2} since CoO and MgO form continuous solid solutions and CoO can be grown readily by the chemical vapor deposition method. Thus, CoO and MgO were co-deposited by two slightly different schemes. In the first case, the standard vertical reactor was modified only to the extent that the CoBr_2 was maintained in a separate crucible at a lower temperature because it was more volatile than the MgBr_2 . The MgBr_2 at 950 C was 4 in. above the CoBr_2 at 800 C, and the seed at 950 C was 1 in. above

the $MgBr_2$. In the second method, $CoBr_2$ and $MgBr_2$ were melted together and appeared to form a molten solution; this solution was used as a single source material and gave Co doped deposits at 935 C. This single source material will be easier to use for long run conditions.

Crystals up to 1 mm thick also were doped with Co by depositing CoO epitaxially on MgO and then allowing the CoO to diffuse into the MgO at 1750 C for 10 days.⁽²⁾ No fluorescence was observed in these crystals in the visible region at room temperature or at 77 K, nor in the IR from 1 μ to 8 μ at room temperature. In MgO , the transmission is negligible beyond 8 μ .

III. CeO_2 GROWTH

A. EXPERIMENTAL

Initial efforts to grow CeO_2 single crystals utilized a multiple-tube flame fusion burner⁽³⁾ with the ultimate temperature of 2800 C⁽⁴⁾. The melting temperature of CeO_2 is listed in the literature as ~2600 C⁽⁵⁾ and 2750 C⁽⁶⁾. A more accurate figure may be higher than either of those listed. As is indicated, a molten cap using the oxy-hydrogen torch might be expected, but none was observed using this torch. At this juncture a higher temperature heat source was sought, and the induction coupled plasma torch was chosen to meet this requirement.

An induction coupled plasma torch was constructed with modifications of Reed's original torch⁽⁷⁾. Initially a 10-kw, 4-mc thermonic r-f generator was used as the power source. As was pointed out in the first quarterly report, the temperatures still were insufficient to melt the CeO_2 sintered preforms readily. Brief overloading of the 10-kw generator indicated the need for more power to obtain a sufficiently hot plasma. A 25-kw generator was available at the North American Science Center for a confirming trial. The desired molten cap was attained and a photo of this was included in the aforementioned quarterly report. A 25-kw, 5-mc Tocco generator was purchased for this facility.

A three-turn, step-wise-wound r-f coil of 3/8 in. copper tubing was made and used with the three concentric tube torch. With approximately 12 kw and an argon-oxygen gas mixture of 30 cfh and 9 cfh respectively for the plasma gas plus helium cooling gas in the outer tube, it was possible to melt a pre-sintered CeO_2 preform. Using Lindsay Code 217 ceric oxide as feed material, average particle size $2.5\ \mu$, and an argon-oxygen mixture for the powder feed gas, polycrystalline growths were made. When the powder feed was stopped and heating continued, or the plasma temperature increased, larger size crystallites were produced in the center of the boule. At the same time the boule length decreased due to some ablation and the high vapor pressure of the CeO_2 . The vapor pressure of the material also created a problem in powder feeding; the small particle sizes appeared to be lost completely when fed through the plasma.

A growth run was made using Lindsay's No. 85 barnesite, the major constituents of which are as follows:

Ceric oxide	45 percent
Lanthanum oxide	22 percent
Neodymium oxide	19 percent
Praesodymium oxide	6 percent
Samarium oxide	3 percent
Gadolinium oxide	2 percent

This material had an average particle size of 3μ . Using a lower power level, but the same gas flow rates as previously mentioned, a good molten cap, as that seen during a sapphire growth, was observed and a small single crystal resulted. The lower melting temperature of this material prevented the previously mentioned vapor pressure problem with CeO_2 .

A powder mixture was prepared which was 10 percent (by weight) La_2O_3 , Lindsay Code 529 (99.997 percent) and 90 percent CeO_2 ; a 17 mol/wt percent La_2O_3 mixture. The material was screened and the -200 mesh particle used as feed powder. Since the melting temperature was appreciably less than 2800 C, the oxy-hydrogen torch was used. The conditions used for the two runs made with this material are as follows:

Run	Pwd Feed Gas O_2 cfh	Torch		Growth Rate in. / hr
		O_2	H_2	
1	6	7	40	0.2
2	2.7	24	40	0.2

The temperature of the caps during growth was determined with an optical pyrometer and observed to be 2250 C. Run No. 1 produced a boule with a dark core. Run No. 2 produced the best-looking crystal growth to date (see Figure 6). The boule unfortunately was broken, as can be seen by the photograph, but visually there appeared to be a single crystal approximately 5 mm long and 2 mm in diameter. A Laue back reflection was made and is shown in Figure 7. This shows that the boule is not single, but is of relatively large grains with some orientation. Petrographic analysis of small chips of the boule showed only internal stresses and no optical activity which would be characteristic of an anisotropic material. Work done by E. Zintl⁽⁸⁾ shows that the fluorite structure is maintained in the CeO_2 - La_2O_3 system with up to 25 mole percent La_2O_3 . We infer, therefore, the material is cubic. A Debye Scherrer pattern (Figure 8) confirms the fluorite type structure of the sample.

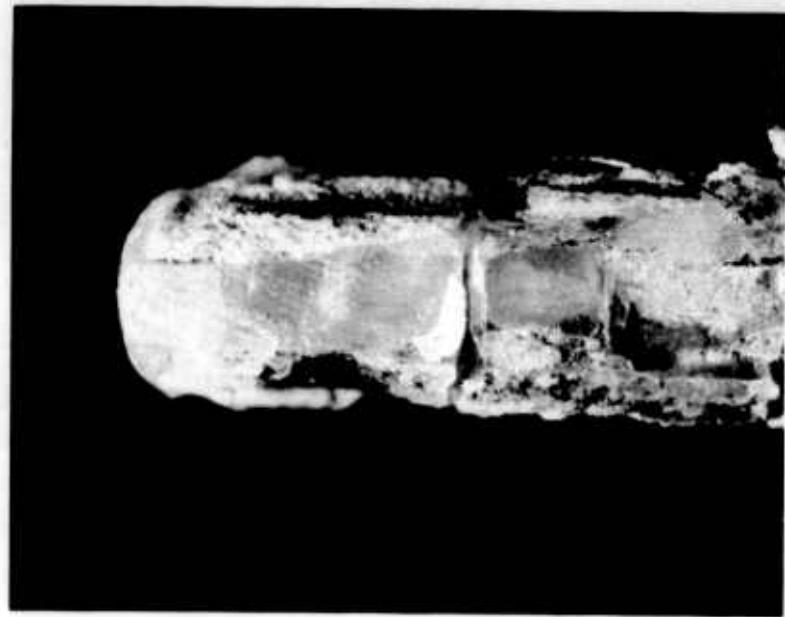


Figure 6. Macrophotograph of Ceric Oxide-Lanthanum Oxide Boule

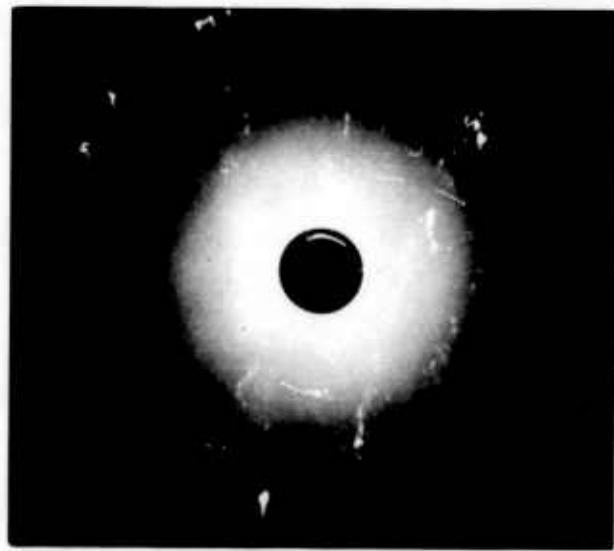


Figure 7. Laue Back Reflection of Ceric Oxide-Lanthanum Oxide Boule

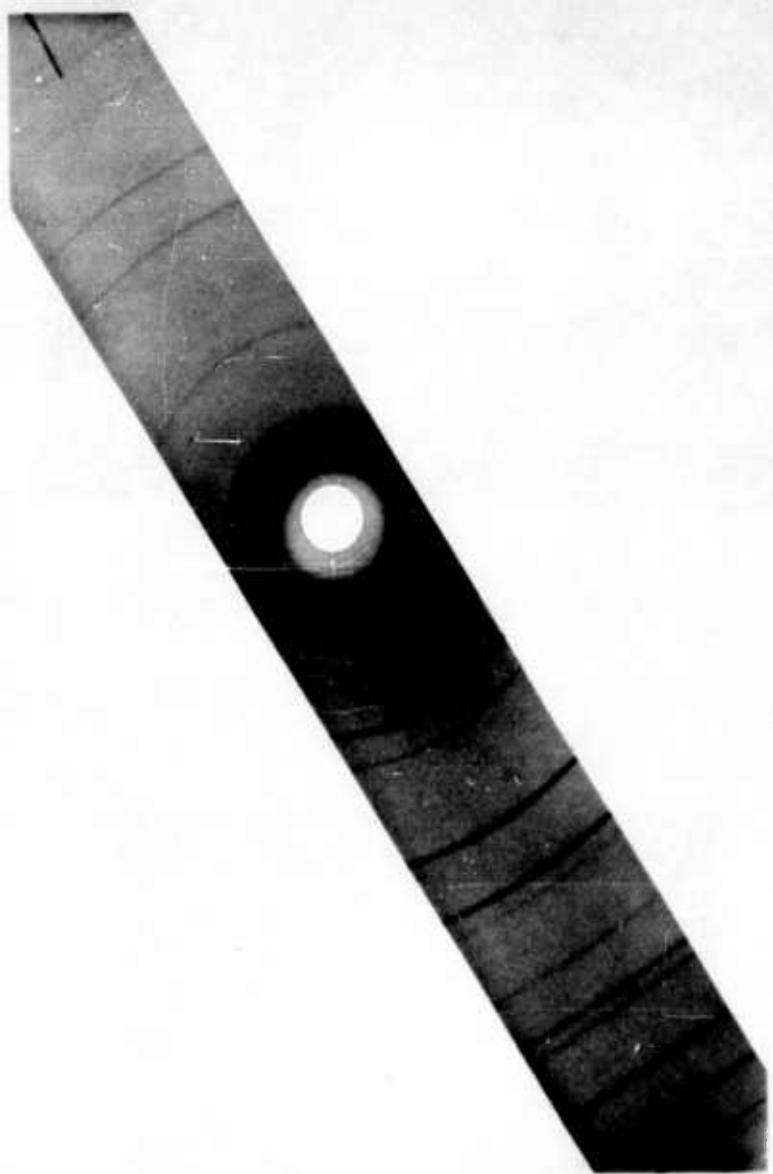


Figure 8. Debye Scherrer Patter of Ceric
Oxide - Lanthanum Oxide Sample

B. SOURCE MATERIALS

The source materials were as reported in the quarterly letter. These were Lindsay Rare Earth Chemical Company's Ceric Hydrate Code 207, Ceric Oxide Code 217, and Code 217 (99. 9+ percent). The other high purity rare earth oxides also were from Lindsay.

Facilities for further purification of these materials are available. Both ionic exchange and repeated precipitation techniques can be used. Lindsay stated that the company is able to supply CeO_2 with 1 to 10 ppm impurities. At such time as the need for higher-purity materials arises, one of the above alternatives will be pursued.

C. SUMMARY

The growth of ceric oxide single crystals has not been satisfactorily achieved at the present time although large crystallites have been obtained. Previous capabilities of the laboratory have been extended in order to obtain usable temperatures in excess of the melting point of cerium oxide (2800 C). Molten caps obtained during the growth process have been observed although these are not as deep as is desired.

Work with the cerium oxide-lanthanum oxide mixtures shows great promise for the growth of large single crystals with the added advantage of growth of a fluorite type structure at lower temperatures.

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IV. FUTURE WORK

Even longer runs will be conducted to grow MgO crystals approaching 1 cu cm in size. Doping studies will be pursued with Cr^{+3} and probably V^{+2} ; long runs then will be made with the most favorable dopant. The effect of higher deposition temperatures on dislocation count will be investigated.

Cerium oxide powders of larger particle size, -100 +200 mesh range have been prepared and are now ready for crystal growth runs using the induction-coupled plasma torch. It is felt that the vapor pressure problem can be minimized with this approach.

Single crystals of cerium oxide-lanthanum oxide will be grown and this will allow doping studies to be initiated at an earlier date. Both the pure cerium oxide and cerium oxide-lanthanum oxide can be pursued simultaneously thus obtaining a larger number of samples for the optical and crystal perfection measurements.

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3	0220	OFFICE, CHIEF, RESEARCH & DEVELOPMENT	54
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3	0225	HUNTSVILLE, ALABAMA	54
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4	0231	ATTN MR. J. W. SMITH		54
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3	0752	U. S. NAVAL ORDNANCE LABORATORY	54
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